AC Electrowetting Actuation of Droplets on a Digital Microfluidic Platform

Isaac Nagiel

Advisor: Dr. Richard Fair
Department of Electrical and Computer Engineering
Pratt School of Engineering
Duke University

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Abstract

As a fairly nascent field, microfluidics is poised to make major strides in the creation of a true lab-on-a-chip platform with applications in the fields of medical diagnostics, drug discovery, and chemical analysis. Due to its short lifetime, research in the field of microfluidics is by no means standardized. Every lab or company engaging in microfluidics has their own proprietary method leading to much division in methodology and practice. One of the most blatant sources of division between different microfluidic labs and companies rests on whether AC or DC voltages should be applied to induce electrowetting. Currently, no formal investigations have been done to conclude which type of actuation is preferential, so this project aims to study two important microfluidic characteristics in an AC vs. DC setting. Purposes of this project include characterizing the effect of frequency on threshold voltage for actuation as well as determining droplet capacitive changes with respect to an applied DC or AC voltage. Threshold voltages were found to be lower for a DC applied voltage only when the frequency was greater than about 150 Hz, but otherwise it was higher. In terms of capacitive effects, applying a DC voltage led to hysteresis when the voltage was stepped up and then down, while an AC applied voltage did not exhibit any hysteresis. These studies provide a solid basis for determining which actuation method is preferential, but further studies are necessary to arrive at a definitive conclusion.

1 Introduction

Microfluidics is an area of research based on the manipulation of small fluid volumes on the order of microliters or nanoliters. More specifically, digital microfluidics refers to the independent manipulation of discrete units of fluid subject to a set of defined operations [1].

The interest in digital microfluidics arises from its tremendous potential to create a true lab-on-a-chip. The goal of a lab-on-a-chip is to create a platform whereby medical diagnostics, environmental monitoring, and basic scientific research can be performed in a rapid, low-cost, portable, and reliable environment [1]. Its tremendous potential is clear due to the low reagent volumes necessary, its reconfigurable nature, and its reduced labor requirement [1].

One of the crucial elements of microfluidics is the electrowetting effect, in which droplets under an applied voltage tend to reduce their contact angle with a surface. A picture showing the electrowetting effect can be found in Figure 1. This wetting allows for various processes to take place on a microfluidic chip including movement, mixing, and splitting. At Duke University, DC actuation is the preferred actuation method, but various other laboratories and companies have moved toward AC actuation [2]. Yet, there hasn't been much research into the question of which actuation method is preferred. Therefore, this project aims to investigate how different actuation methods affect the wetting properties of a droplet.

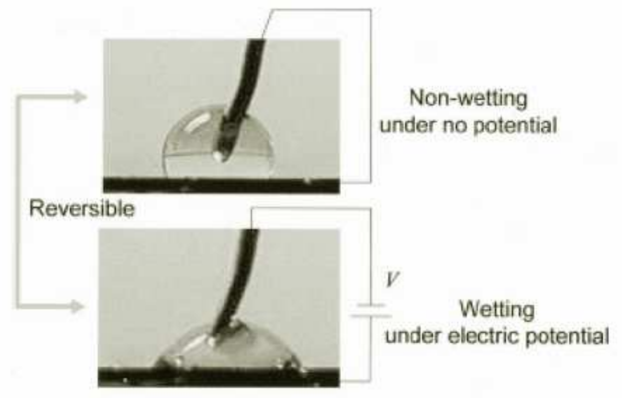


Figure 1: The electrowetting effect

1.1 Digital Microfluidic Chip

A polarizable and conductive liquid is placed between two plates as shown in the cross-section view of a digital microfluidic chip in Figure 2. The droplet is immersed in oil, which allows for actuation under lower voltages. Above the droplet is a continuous ground top-plate made of glass, which is first coated with Indium Tin Oxide (ITO) to make the surface conductive and then coated with Teflon AF (∼60nm) to make the surface hydrophobic. Below the droplet is another glass slide which is coated first with Parylene (∼800nm), which serves as a dielectric and then with Teflon AF (∼60nm) to make the surface hydrophobic. Implanted within the glass and beneath the dielectric are electrodes. These electrodes have traces attached to them, which are connected to contact pads that allow for voltages to be applied to the electrodes.

A sample microfluidic chip can be seen in Figure 3. The green that can be seen is the gasket layer. This gasket layer creates spacing between the bottom surface of the chip and the ground plane (not shown) thereby eliminating the possible of a short occurring. The large

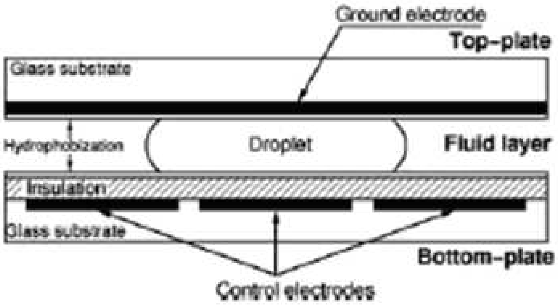


Figure 2: Cross section of a digital microfluidic chip

pad on the center-top of the chip is the contact ground contact pad allowing for a connection between the ground plane and the ground of the power supply. Due to the immense amount of electrodes, the electrodes are connected in a bus fashion, so every 4th track electrode on one half of the chip corresponds to the same contact pad.

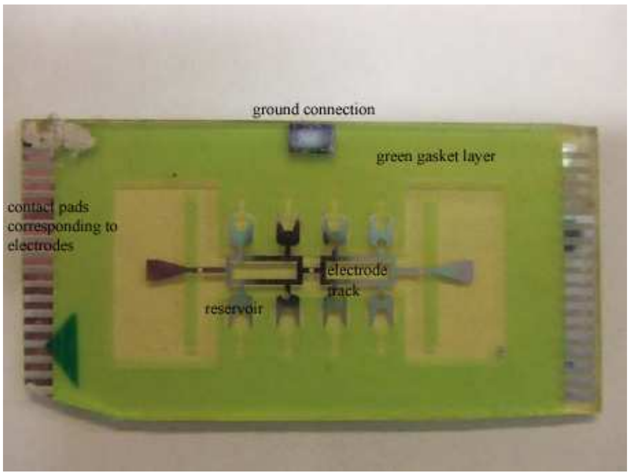


Figure 3: Sample microfluidic chip

2 Voltage Threshold Comparison

2.1 Motivation

Researchers in the field of microfluidics are always trying to figure out ways to reduce the threshold voltage required to actuate the droplet. So far, the lowest threshold voltages achieved using electrowetting has been on the range of 15-20 V [2]. Yet, for the purpose of the creation of a portable lab-on-a-chip device, this voltage range is too high. Ideally the threshold voltage would be compatible with that of a easily obtainable household battery. Another issue is that continued actuation with such high voltage tends to lead to degradation of the insulator, which will reduce the reliability of the microfluidic device [2]. Therefore, threshold voltage seems like an obvious place to start when doing an analysis comparing the DC and AC actuation methods.

2.2 Experimental Setup

A microfluidic chip of the same design as shown in Figure 3 was mounted onto a stage where an electronic test clip was used to contact the pads along the sides of the chip. The electronic test clip connected to a computer-controlled test unit that was capable of switching up to 50 outputs between ground and the voltage applied by either the DC Agilent 3612A power supply or Agilent 33250A waveform generator. The Agilent 33250A was connected in series with the FLC Electronics' F10A Voltage Amplifier, which allowed for higher voltages to be applied since the AC source was constricted to 10 Vpp. A top-plate with holes above the reservoirs was then placed directly on top of the chip and kept in place by four alligator clips.

2.3 Experimental Results

Using a pipet, 2 cSt silicon oil was inserted between the bottom and top layers of the chip. A .1 M KCl droplet was then dispensed from a reservoir and moved to a location of the chip where there was a series of four electrodes in a row. The controller was programmed to move the droplet back and forth along the four electrode series. The DC voltage was increased until the droplet could easily track the 4 electrode sequence. Once full tracking was determined, the voltage was reduced until tracking became sluggish. This was determined to be the DC threshold voltage. Then, the DC voltage was disconnected from the controller and the AC source was connected. The same procedure was applied for the AC source by controlling the peak-to-peak voltage on the output. Various frequencies were also applied in order to determine the effect of frequency on threshold voltage.

A comparison of the same droplet and same set of four electrodes was used for each independent trial. Results from 3 trials can be found in Figure 4. All three trials had DC actuation voltages between 25 and 30 Volts. They also all exhibited a drop in threshold voltage at about 10 Hz frequency followed by an increase in threshold voltage as the frequency increased. In all three cases, the 1 KHz frequency had the highest actuation voltage, yet this actuation voltage differed from the DC threshold by less than 5 Volts in all three cases.

2.4 Discussion

The experimental results clearly show that frequency indeed has an effect on the threshold voltage needed for actuation. From the data, the lowest actuation voltages come from AC actuation where the frequency is very small, around 10 Hz. Jones attempted to explain this effect by claiming that DC electric fields, unlike AC, don't provide any time-varying force components that help overcome wetting-related stiction effects [4]. Yet, once the frequency

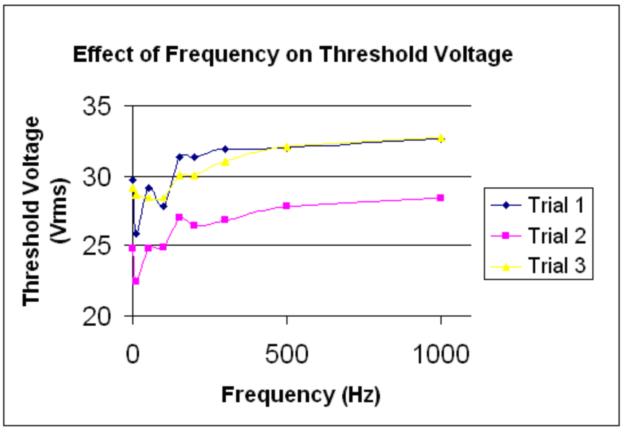


Figure 4: Frequency effects on threshold voltage

is increased considerably, the DC actuation scenario seems to be preferable due to its lower threshold voltage.

3 Capacitive Effects

3.1 Motivation

One of the most undesirable aspects of the microfluidic platform is its reduced reliability over time. In order to create a true lab-on-a-chip, it is necessary to create a chip that will work with the same effectiveness whether it be the first trial or the 200th trial. The repercussions of a faulty chip are immense when used in a medical diagnostic setting. Initial hypotheses into the lack of reliability maintained the possibility that with the application of the electric field, the insulator layer was becoming charged. This suggested that higher voltages would need to be applied as the insulator became charged in order to produce equivalent wetting. Therefore, a capacitance measurement apparatus was constructed to investigate the insulator charging hypothesis.

3.2 Experimental Setup

A ring-oscillator circuit in Figure 5 designed by Michael Pollack et al. was used to measure the change in period due to the electrowetting effect. The circuit was calibrated using known capacitances in order to develop a capacitance vs. period curve found in Figure 6. The period was measured using a data acquisition card, which sampled at a rate of about 400 times a second. This data was converted to a capacitance by setting a trend line on the calibration curve in order to get an equation for the capacitance-period relationship. This conversion was done in LabView and the process can be seen in Figure 7. LabView, using GPIB technology, also controlled the AC and DC power supplies in order to step up and step down the voltage applied. The DC power supply was the Agilent E3631A. The AC source was the Agilent

33250A waveform generator connected to FLC Electronics' F10A Voltage Amplifier, which allowed for higher voltages to be applied since the AC source was constricted to 10 Vpp. The capacitance measurements were then fed to an Excel spreadsheet.

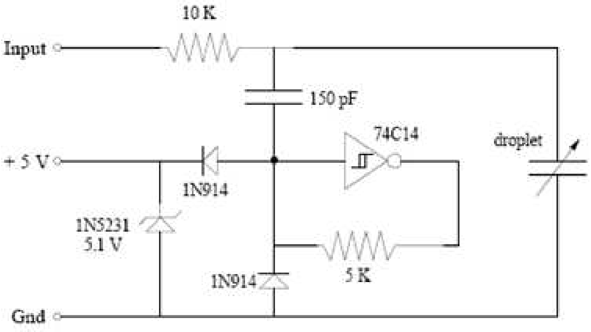


Figure 5: Ring-oscillator circuit used for capacitance measurements

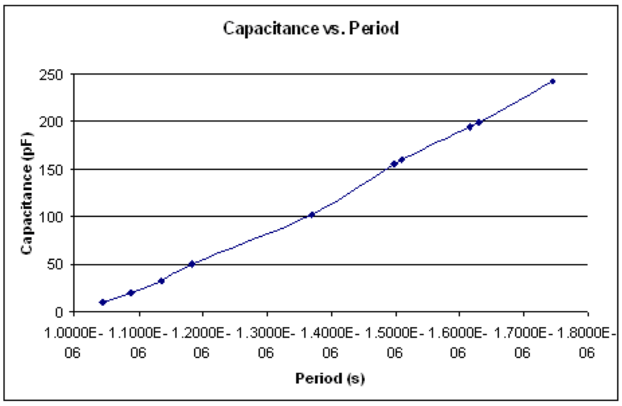
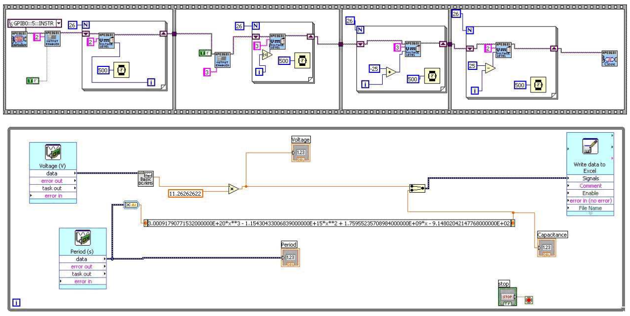


Figure 6: Calibration curve



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3.0009179077153200000E+20*x**3 - 1.1543043300683900000E+15*x**2 + 1.7595523570898400000E+09*x - 9.1480204214776800000E+02
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Figure 7: LabView block diagram used to gather capacitance and voltage data

3.3 Experimental Results

3.3.1 Capacitance Experiments with no Oil

A 5 μL .1 M KCl droplet was placed on an electrode in which the droplet is smaller than the electrode. This allows for maximum spreading of the droplet as it is not constricted at all by the size of the electrode. A probe set as ground was inserted into the top of the droplet until it reached about $\frac{1}{4}$ of the way down into the droplet. A voltage step sequence was then applied to this system to be able to see the effect of voltage on capacitance. This experiment allowed for variation in voltage step time, size of voltage step, and final voltage reached. Figure 8 shows the capacitance-voltage curve for a 50 V terminal voltage with a 1 s interval and a 1 V step size. The increased voltage leads to a greater capacitance as the droplet wets the surface. This wetting leads to increased spreading of the droplet, which creates a greater surface area for the droplet at the interface with the insulator.

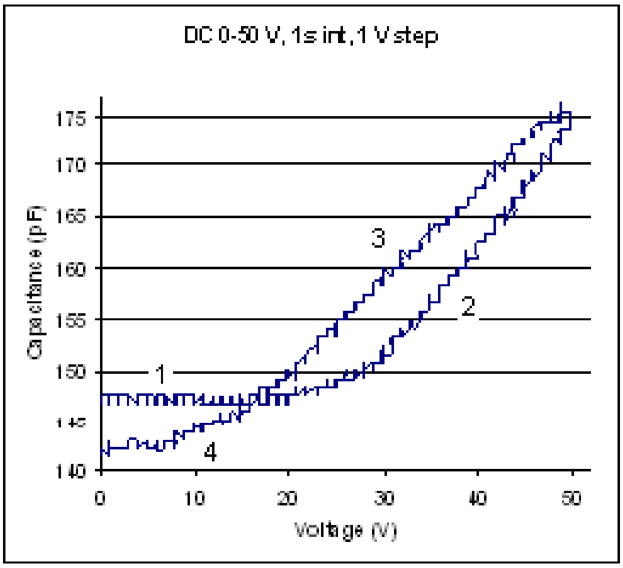

Figure 8: Voltage hysteresis curve with path sequence 1,2,3,4

It is interesting to note on Figure 8 the hysteresis that occurs when a DC voltage is applied. As the voltage is stepped up then down, the capacitance loop goes in the path 1,2,3,4. On the return path, for the same applied voltage, at voltages greater than about 18 V, there is a larger capacitance. This phenomenon does not occur when AC actuation is applied as can be seen by Figure 9. Figure 9 represents data from the same experimental setup as in Figure 8 with one key difference, which is the use of an AC source inducing electrowetting of the droplet. Thus, the lack of hysteresis under AC actuation is an interesting feature of microfluidics. Repeated measurements show the same hysteresis occurring only for applied DC voltages, while absent under the AC bias.

Other experimentation included seeing the effects of decreased time intervals between voltage steps as well as the effect of frequency. The effect of decreasing interval time to 250

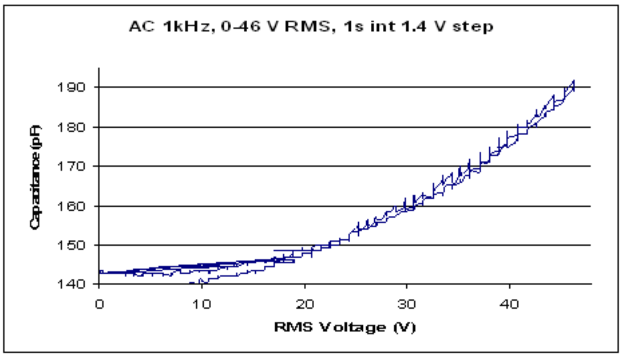

Figure 9: AC actuated capacitance vs. voltage curve

ms as opposed to 1 s led to a decreased capacitance at the maximum of 50 V bias, as perhaps the system did not have enough time to respond to the voltage steps. Also, one can note that the starting and ending points of the capacitance values are at about the same value as opposed to Figure 8. This is most likely due to evaporation of the droplet. Since the droplet in Figure 10 is exposed for a quarter of the time that the droplet of Figure 8 is exposed, there is much less evaporation. An evaporation curve showing how the droplet capacitance decreases under no external voltage bias is shown in Figure 11. This makes sense as the surface area of the droplet at the insulator interface decreases as the droplet reduces in size due to evaporation. The effect of small changes in frequency on the droplet is also not too noticeable in terms of wetting or hysteresis as shown in Figure 12.

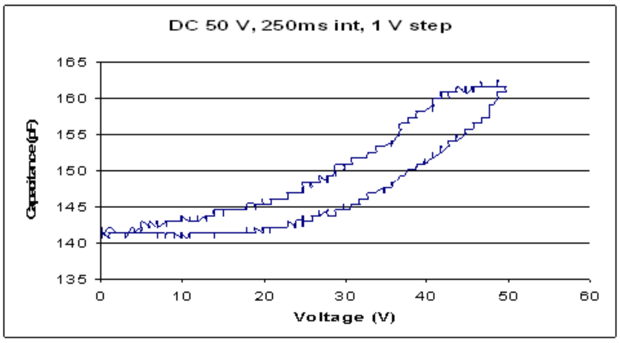

Figure 10: DC hysteresis curve with shortened voltage step interval time

Another question is whether increasing the voltage can somehow induce hysteresis in the AC actuation case. Figure 13 has an increased terminal voltage and yet there still is no hysteresis present.

3.3.2 Capacitance Experiments with Oil

When the droplet was immersed in 5 cSt oil as in Figure 14, no significant differences in behavior could be seen in the voltage-capacitance curves. The hysteresis still existed, yet the

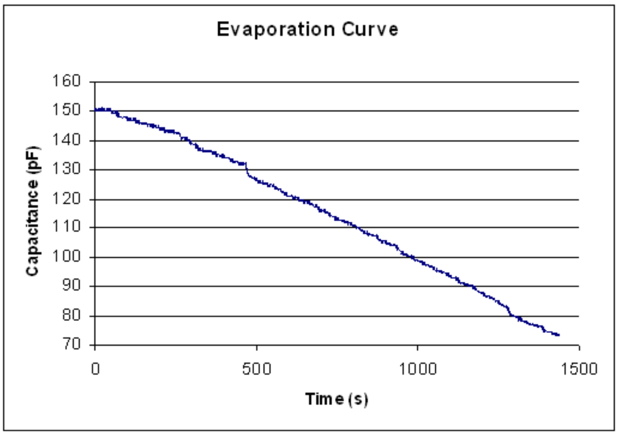

Figure 11: Evaporation of the droplet over time

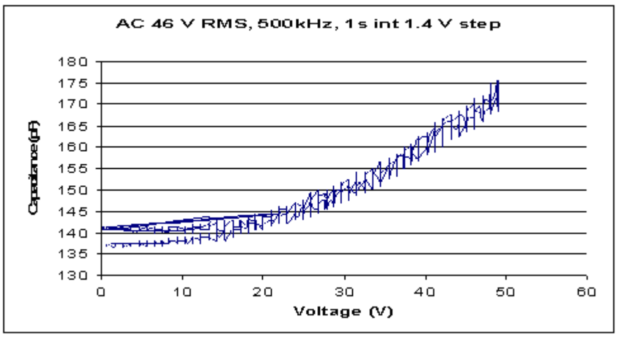

Figure 12: 500 kHz frequency AC actuation curve

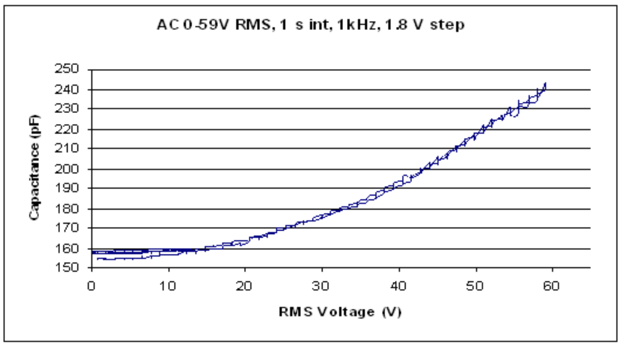

Figure 13: Hysteresis still not present at higher AC actuation voltage

evaporation wasn't present as can be seen that the starting and ending values of capacitance are about equal. Subsequent AC actuation experiments also showed no significant differences when the droplet was in the oil medium. In this no top-plate setup, the oil did not seem to affect the capacitance values over time either, which was contradictory to the results noted in a top-plate setup experiment[3]. This can be seen as the static capacitance remains basically

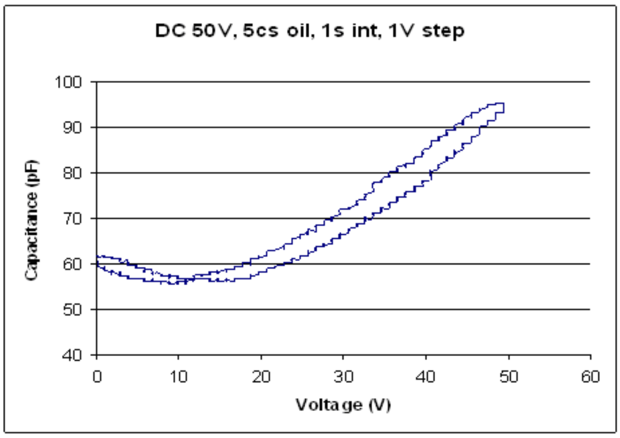

Figure 14: DC hysteresis curve with droplet immersed in oil

constant with respect to time under a constant 50 V bias in oil as shown in Figure 15. Different viscosity oils such as 1 and 2 cSt demonstrated the same effects as in Figure 15.

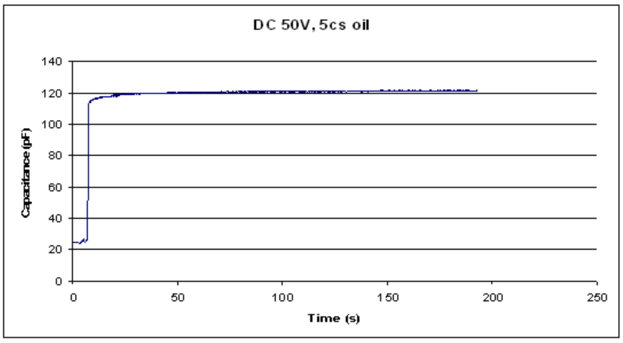

Figure 15: Effect of oil on droplet wetting over time with constant DC voltage

3.4 Discussion

The hysteresis shown under a DC bias is a real and reproducible effect. It's most likely cause is due to ”sticky” polarization within the insulator material, which doesn't occur when an AC bias is applied. As can be seen by Figure 16, which is a plot of polarization against applied electric field for a ferroelectric material, remnant polarization occurs as the polarization is non-zero at negligent electric field on the path back. This evidence shows how the dipoles remained lined up causing negative charge to stay at the insulator interface with the liquid thereby inducing wetting. A diagram of this can be seen in Figure 17. This wetting increases the capacitance and explains why the hysteresis occurs for a DC source. The hysteresis doesn't occur under AC as the polarity is switching so quickly that the dipoles do not have time to ”stick” in a certain alignment.

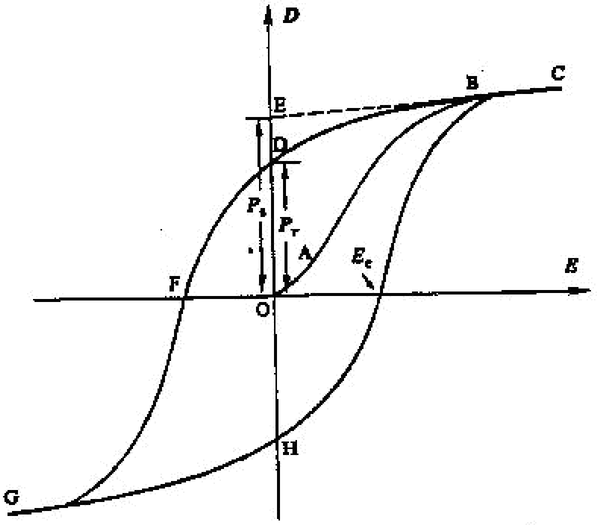

Figure 16: Plot of polarization against applied electric field for ferroelectric material[5]

The oil in the no top-plate setup seems not to have much effect on the capacitance under applied voltages other than to reduce the evaporation to an almost negligible level. Experimentation into the oil's effect with a top-plate is necessary to better characterize its effects. Recent experiments suggest there is some interaction between the oil and the droplet in a top-plate setup but further experiments are necessary.

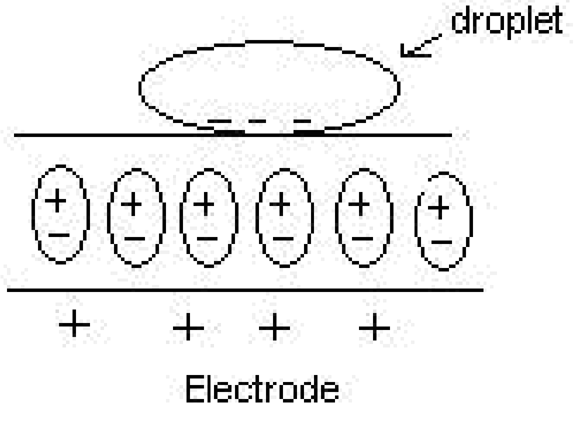

Figure 17: Diagram of effect of dipole polarization on wetting

4 Conclusion

This investigation has a limited scope in trying to tackle the larger and more complex question of which actuation method is preferable. Yet, it provides valuable insight into two of the most interesting aspects concerning the future and stability of microfluidics. It delves into the issue of reliability and threshold voltage. Results have shown that at very low frequency such as 10 Hz, one can get minimal threshold voltage. Yet, as the frequency increased, DC actuation became the preferred actuation method due to its lower threshold voltage. In terms of capacitive effects, there is an obvious hysteresis that occurs whenever DC voltages are applied to the droplet. This is most likely due to remnant polarization within the dielectric layer inducing increased wetting of the droplet. To this end, it seems that AC actuation would be preferred in that it restricts the hysteresis effects allowing for a more reliable system. In other words, a user of the microfluidic system will always be able to associate a given amount of wetting with a certain voltage without having to let the system rest so dipoles can return to their relaxed state. Therefore, it seems that AC actuation would be the preferred method as it exhibits no hysteresis and only minimal increases in threshold voltage at higher frequencies. Yet, further research into many of the other aspects of microfluidics is still necessary to come to a definitive conclusion.

5 Future Projects

Further projects in this field include extensive trials with top-plates including characterizing the effects of oil in the system. Initial studies show that under a constant voltage, the capacitance of the droplet in air remains fairly constant. Yet, when the droplet is immersed in oil, there seems to be a drop in capacitance over time. Therefore, oil must be causing this decrease in capacitance because it is the only variable that is changed between the two experiments. Initial hypotheses include that when the bias is applied, the droplet spreads and gets rid of all the oil between it and the insulator surface. Then as time goes by and the system reaches an equilibrium, the oil seeps back under the droplet thereby decreasing the capacitance. It would also be interesting to note how different viscosity oils affect how the interaction between the droplet and the oil take place.

6 Acknowledgements

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